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IMPACT OF NITRILOTRIACETIC ACID (NTA) ON AN ACTIVATED SLUDGE PLANT - A FIELD STUDY

by

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ABSTRACT

A field study on the effects of nitrilotriacetic acid (NTA) on activated sludge was carried out at the Waterdown Vater Pollution Control Plant, from January, 1972 to April, 1973. The Waterdown Plant is a conventional activated sludge plant with a design flow of 1 362 m³/d. The nominal wastewater flow was approximately 45% of design capacity and all homes served by the sewer system were within a mile and a half of the treatment plant.

The areas investigated in the study include: degradation of NTA, effect of NTA loading on chemical treatment for phosphorus removal, NTA loading and heavy metal removal in the treatment plant, and effect of NTA loading on treatment plant operation.

The NTA was added to the treatment plant in the form of Sunlight Soap (a commercial laundry detergent containing 20 wt % NTA) at two rates to approximate NTA spiking levels of 8 mg/L NTA and 16 mg/L NTA. The background NTA loading in the raw wastewater averaged 2.5 mg/L NTA. Diurnal variation studies of the NTA loading showed peak values of 12 mg/L NTA in the afternoon and minimum values of 0.1 mg/L NTA at night. The raw wastewater had an average BOD_5 of 100 mg/L, a suspended solids of 150 mg/L, and a total phosphorus loading of 6.0 mg/L P. Heavy metal levels averaged 0.2 to 0.5 mg/L for zinc, aluminum and iron, and 0.01 to 0.1 mg/L for lead, nickel and copper. The treatment plant efficiency averaged 86% BOD_5 and 85% suspended solids removal throughout the study.

Such factors as microorganism acclimatization to NTA, chemical addition for P removal, temperature and NTA loading, were analyzed by grouping the field data in such a manner that the sole effect of each factor could be analyzed with all other factors held constant. The studies demonstrated that NTA loadings up to 16 mg/L NTA did not adversely affect treatment plant efficiency or the amount of chemical precipitant required to meet an effluent total phosphorus objective of 1 mg/L P. NTA degradation was significantly affected by wastewater temperature. Correlation of heavy metal transport through the treatment plant with NTA loading was inconclusive.

RÉSUMÉ

Les effets de l'acide nitrilotriacétique (NTA) sur les boues activées ont fait l'objet d'une étude expérimentale, de janvier 1972 à avril 1973, à l'usine d'épuration de Waterdown. Il s'agit d'une installation classique aux boues activées, d'une capacité de 1362 m³/j. Cette capacité n'est exploitée qu'à 45% environ. Toutes les habitations desservies par le réseau d'égout sont situées dans un rayon d'un mille et demi de l'usine.

Les points étudiés ont été la dégradation du NTA, les effets de sa charge sur la déphosphatation, sur l'élimination des métaux lourds et sur le rendement de l'épuration.

Du savon Sunlight, qui contient en poids 20% de NTA, a été ajouté en quantité suffisante pour que la teneur en NTA atteigne 8 et 16 mg/L. Avant ces additions, la concentration de NTA dans les eaux brutes était de 2.5 mg/L. D'après les études de la variation, sur 24 heures de la charge de NTA, sa concentration maximale (12 mg/L) a été atteinte l'après-midi, tandis qu'elle était à son minimum (1 mg/L) la nuit. En moyenne, les eaux brutes avaient une DBO₅ de 100 mg/L, 150 mg de matières en suspension/L et 6.0 mg P/L. Leur teneur en zinc, en aluminium et en fer variait entre 0.2 et 0.5 mg/L, celle du plomb, du nickel et du cuivre, entre 0.01 et 0.1 mg/L. Au cours de l'étude, la DBO₅ a été réduite de 86% en moyenne, et 85% des matières en suspension ont été éliminées.

L'analyse de l'adaptation des micro-organismes au NTA, de l'addition de réactifs pour la déphosphatation, de la température et de la charge de NTA s'est faite à partir de données expérimentales que avaient été regroupées de telle façon qu'on puisse faire varier un seul facteur à la fois, les autres demeurant constants. Les recherches ont montré que des charges de NTA aussi élevées que 16 mg/L n'ont pas nui à l'épuration ni nécessité davantage de réactifs chimiques pour atteindre l'objectif de l mg de P/L dans l'effluent. La température des eaux résiduaires a influé sensiblement sur la dégradation du NTA. Aucune corrélation n'a pu être établie entre la circulation de métaux lourds et la charge de NTA.

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CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

A field study on the effects of nitrilotriacetic acid (NTA) was carried out at the Waterdown Water Pollution Control Plant from January, 1972 to April, 1973. The areas investigated in the study included: NTA degradation, the effect of NTA loading on chemical treatment for phosphorus removal, and NTA loading and heavy metal removal. The studies demonstrated the following:

- NTA loadings up to 16 mg/L NTA did not adversely affect the amount of chemical precipitant required to meet an effluent total phosphorus standard of 1 mg/L P. Chemical treatment processes included both alum addition to the aeration tank, and ferric chloride and polymer addition to raw sewage.
- NTA loadings up to 16 mg/L NTA did not adversely effect the treatment plant efficiency or cause any operating problems.
- The microbial population in the aeration tank required approximately two to three weeks to acclimatize to each of the higher NTA loadings.
- 4. NTA degradation in the treatment plant was significantly affected by wastewater temperature. With background NTA levels of 3 mg/L NTA or less, the degradation rate was relatively independent of wastewater temperature. With high NTA loadings (NTA levels greater than 8 mg/L NTA) the removal rate was strongly correlated with wastewater temperature. The NTA removal rate also decreased with increased NTA loading.
- The correlation of heavy metal transport through the treatment plant with NTA loading was inconclusive. Zinc and iron removals in the treatment plant decreased with increasing NTA concentration in the effluent while copper, aluminum, nickel and lead showed no correlation.

RECOMMENDATIONS

The following specific areas for further investigation are recommended:

- 1. A detailed study of the removal of NTA from a large sewage treatment plant [45 400 m³/d (10 MIGD)] should be undertaken. The performance during low temperature winter conditions should receive close scrutiny. The plant chosen should also have a relatively high metals input so that metal transport may also be monitored.
- 2. More fundamental studies at pilot plant level where close control can be maintained are required to evaluate the effects of temperature, NTA loading and NTA shock loading on NTA removal by the activated sludge process. Controlled experiments into the effect of NTA loading on metal transport are also necessary to elucidate the metal transport mechanism.

INTRODUCTION

I

In August, 1971, the government of Canada and the government of the province of Ontario signed an agreement to ensure that the water quality of the Great Lakes is restored and protected. This "Canada-Ontario Agreement on Great Lakes Water Quality" was signed in response to the recommendations of the International Joint Commission (IJC) concerning pollution of the Lower Great Lakes and in anticipation of the Canada-United States Agreement on Great Lakes Water Quality. The purpose of this Canada-Ontario Agreement was to permit Canada and Ontario to effectively carry out their obligations under the International Agreement. An additional important provision of the Agreement was for the conduct of a research program for reducing costs of programs to achieve the specific water quality objectives set out in the Agreement. Thus, late in 1971, research programs were initiated on chemical removal methods, sludge handling, sludge disposal and other matters related to the process of removal of nutrients such as phosphorus from sewage.

In May, 1972, the Federal Environment Minister announced new regulations, effective January, 1973, further restricting the phosphorus content of detergents to 5% P_2O_5 . At the same time, the Minister also announced that the levels of substitute builders would be monitored to safeguard the environment. One such substitute builder is nitrilotriacetic acid (NTA) which has been used in some synthetic detergents as a substitute for polyphosphates which are considered to be a major cause of lake eutrophication. However, questions have been raised as to the possible environmental effects that could result from a large scale usage of NTA in detergents.

As a result, many laboratory studies have been conducted on the biodegradability of NTA and its metal complexes (Shannon et al, 1978) and their toxicity to marine life. Most of these studies are summarized in the comprehensive literature reviews of Thom (1971), Epstein (1972), Thayer and Kensler (1973) and Prakash (1976). There

was also a full scale field study on NTA removal by the activated sludge process (Shumate et al, 1970). During the winter of 1971, a full scale field study on the effect of NTA detergent on a biological treatment plant was carried out by the Environmental Protection Service at the Canadian Forces Station Gloucester (Shannon and Kamp, 1973). A similar study was conducted by the Fisheries Research Board at two aerated sewage lagoons near Winnipeq, Manitoba (Rudd and Hamilton, 1972).

1.1 Objectives

In light of the possible environmental effects of NTA on treatment plant operation and the receiving waters, the following areas were investigated in the laboratory and under full scale conditions during the winter of 1972/1973:

- Nominal background levels of NTA in sewage treatment plant influent and effluent.
- 2. Degradation of NTA in an activated sludge treatment plant.
- Effect of NTA loading on the chemical precipitation of phosphorus in a sewage treatment plant.
- Effect of NTA loading on heavy metal removal in a sewage treatment plant.
- 5. Degradation of NTA in receiving streams.

The test site of the study was the Waterdown Water Pollution Control Plant. The degradation of NTA in receiving streams has already been reported by Shannon et al (1974).

2 STUDY SITE AND EXPERIMENTAL PROCEDURES

2.1 Plant Description

The field test site for the study was the Waterdown Water Pollution Control Plant built in 1965 as a conventional activated sludge plant with a design flow of 1 362 $\rm m^3/d$ (0.3 MIGD). The nominal wastewater flow was approximately 45% of design capacity and all homes served by the sewer system are within a mile and a half of the treatment plant. A schematic diagram of the plant is shown in Figure 1, with design details summarized in Table 1. Throughout the study, the two secondary

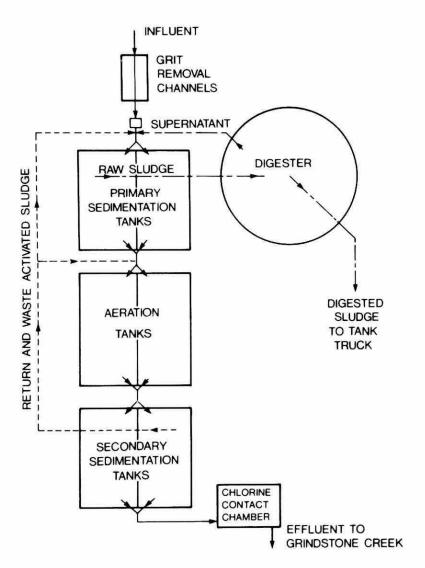


FIGURE 1. WATERDOWN WATER POLLUTION CONTROL PLANT

sedimentation tanks remained in operation, however, one primary clarifier and one aeration tank were taken out of service. This configuration most closely approached design conditions for the nominal wastewater flow. The effluent from the plant is discharged to Grindstone Creek and eventually finds its way to Hamilton Harbour.

TABLE 1. WATERDOWN WATER POLLUTION CONTROL PLANT DESIGN DATA. DESIGN FLOW = 1362 m3/d (0.3 MIGD)

Process		Uni	t Process
Conv. Act. Sludge	Identification		Description
E S	Screening	Type : Size :	Manually cleaned One, 2" spacing
REAT	Grit Removal	Type :	Channel, manually cleaned
PRIMARY TREATHENT	Sedimentation	Type : Size : Retention : Loading :	Walker Process CRP Two, 30' x 8' x 8' (24 000 gal) 1.9 hours Surface 625 gal/ft ² /d Weir 6 520 gal/ft/d
5	Aeration		
(BIOLOGICAL) TREATMENT	Tanks	Size :	Diffused air, single-pass Two, 30' x 14' x 11' (96 000 gal) 7.7 hours
GICAL)	Air Supply		Aerzener Blowers Two, 600 cfm
(81010)	Diffusers	Type : Spacing :	Chicago Pump Discfusers 48 (total) @ 2' centres
SECONDARY	Sedimentation	Type : Size : Retention : Loading :	Two, 30' x 8' x 11' (33 000 gal) 2.6 hours
DISIN- FECTION	Chlorination	Contactor :	One, F & P 2-40 lb/d 6' x 17' x 8'-9' (5 600 gal) 27 minutes
SLUDGE HANDL ING	Digestion		Anaerobic, fixed steel cover mixed by recirculation, single stage One, 30' dia x 17' swd (94 000 gal)

Note: Multiply - ft \times 0.304 = m, gal \times 0.00454 = m², lb/d \times 0.454 = kg/d, gal/ft/d \times 0.454 = m/m-d, oal/ft²/d \times 0.049 = m/m-d, oal/ft²/d \times 0.0491 = m²/m²d.

2.2 Experimental Program

The experimental program consisted of the following study conditions during which different chemicals were added to various sewage streams at a number of NTA loadings:

- (1) Laboratory study jar tests concerning the effect of NTA on phosphorus removal.
- (2) Field study aluminum sulphate and NTA addition period.

- (3) Field study ferric chloride, polymer (Dow A-23) and NTA addition period.
- (4) Field study NTA addition period.

A listing of the field study conditions in chronological order is given in Table 2.

TABLE 2. EXPERIMENTAL FIELD PROGRAM AT WATERDOWN

Field Study Conditions	NTA Spiking Levels ¹	Date	Average NTA Conc. As H ₃ NTA	Average Temp. °C
Baseline	NTA-0, No Alum	Jan 10 - Jan 21	3.0	- 11
Alum	NTA-0	Jan 24 - Feb 11		9
Addition	NTA-8	Feb 14 - Mar 6		10
	NTA-16	Mar 7 - Mar 24	14.5	9
	NTA-0	Apr 11 - Apr 28	1.2	11
	NTA-8	May 1 - May 11		12
FeC1;	NTA-16	May 15 - May 26		15
Addition	NTA-0.	The water the state of the stat	1000000	1,170
	No FeCl3	May 30 - June 7	2.3	16
	NTA-0	Nov 6 - Nov 19	2.0	15
NTA	NTA-8	Dec 4 - Dec 17		12
Addition	NTA-16	Jan 1 - Jan 14		10
1040	NTA-0	Jan 29 - Feb 11	2.6	10
NTA	NTA-8	Feb 26 - Mar 11		10
Addition	NTA-16	Mar 26 - Apr 5		10

¹ In mg/L.

2.3 Field Procedures

Nitrilotriacetic acid was added to the raw sewage in the form of Sunlight Soap, a commercial laundry detergent. The stock soap solution was made up every morning by adding 0.125 kg of soap per litre of tap water. A constant daily pumping rate was set each morning based upon the raw sewage flow of the previous day and the observed instantaneous flow between 0800 and 0100 hours. The soap solution was added to a manhole approximately 100 m (300 ft) upstream from the head end of the grit channel.

Two chemical types and addition points were used for phosphorus removal during the study. In the first, simultaneous chemical treatment was employed by adding alum to the mixed liquor in the inlet channel to the final clarifier, such that a concentration of 200 mg/L alum was present. The precipitated phosphates were removed along with the activated sludge in the final clarifier. During the second phase,

phosphorus removal was accomplished by chemical treatment of raw sewage. Ferric chloride at 30 mg/L Fe³⁺ was added to the influent end of the grit removal channel followed by an anionic polymer (Dow A-23) at 0.5 mg/L to the Parshall Flume. The precipitated phosphates were removed with the particulates in the primary clarifier. A constant daily pumping rate (for these chemicals) was determined using the method developed for the soap solution. The chemical stock solutions were made up as follows:

- 1. Ferric chloride 300 g/L.
- 2. Alum 640 g/L.
- 3. Purifloc A-23 polymer 2.5 g/L.

2.4 Field Sampling Program

Seven points within the Waterdown Water Pollution Control Plant, as shown in Figure 2, were selected as sampling stations: raw sewage main (S1), effluent channel secondary clarifiers (S2), effluent channel primary clarifiers (S3), aeration tank (S4), return activated sludge overflow box (S_5) , raw sludge pump (S_6) , and the supernatant return line (S_7) . Automatic samplers were set up for the duration of each study period at S1, S2 and S3. Hourly samples were taken continuously for the sampling period, 24 hours a day. Eight-hour composites were prepared from these hourly samples for the following three time periods: 0100 to 0800 hours, 0900 to 1600 hours, and 1700 to 2400 hours. In the final NTA study period, six-hour composites were prepared from the hourly samples for the following four time periods: 0100 to 0600 hours, 0700 to 1200 hours, 1300 to 1800 hours and 1900 to 2400 hours. The resulting data from these samples were used to determine phosphorus removal and NTA degradation efficiencies measured. Twenty-four-hour composites were also prepared from these hourly samples. They were used to assess plant performance by analyzing for total BOD5, suspended solids, volatile suspended solids, and soluble total organic carbon. They were also used to measure heavy metal transport by analyzing for aluminum, iron, copper, zinc and nickel. Grab samples of other streams

were taken in order to gain a more complete understanding of the system. They included: daily grab samples of the aeration tank contents for mixed liquor suspended solids, mixed liquor volatile suspended solids, sludge volume index and total BOD_5 plus daily grab samples of the waste activated sludge, raw primary sludge and digester supernatant for suspended solids, volatile suspended solids, iron, aluminum and total phosphorus.

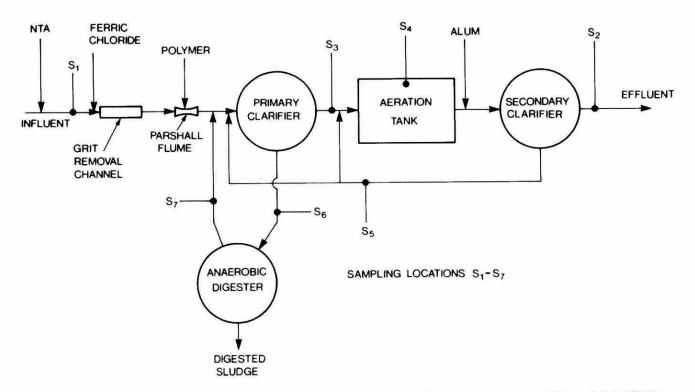


FIGURE 2. TREATMENT PLANT SAMPLING AND CHEMICAL ADDITION LOCATIONS

All routine samples were transported daily to the Wastewater Technology Centre, Burlington, for analyses. Special preservation techniques and analyses were required for some samples, and these are described in a later section.

2.5 Jar Testing Procedures

One of the objectives of the study was to assess the relationship between NTA loading and chemical phosphorus removal. For this investigation standard jar testing procedures using Phipps and Bird six-place gang stirrers were used for the three different phosphorus removal coagulants (ferric chloride, lime and aluminum sulphate). A series of jar tests were conducted on samples from the influent and effluent streams of several wastewater treatment plants located in the Burlington area. The source of the samples and number of jar tests is shown in Table 3. After the samples were brought to the laboratory, they were spiked to the required level of 8 and 16 mg/L of NTA by adding the sodium salt of NTA. The ranges of chemical doses tested were as follows: lime, 0 to 500 mg/L as Ca(OH)₂; ferric chloride, 0 to 30 mg/L as Fe³⁺; and alum, 0 to 30 mg/L as Al³⁺. The anionic polymer (Purifloc A-23, Dow Chemical Company) was used as a coagulant aid at 0.5 mg/L with ferric chloride and alum.

TABLE 3. SOURCES OF SEWAGE FOR JAR
TESTS TO ASSESS NTA LOADING
AND CHEMICAL PHOSPHORUS
REMOVAL

Waste Treatment Plant	Sample Type	No. of Jar Tests
Waterdown	Raw Sewage	72
	Secondary Effluent	23
Skyway Plant	Raw Sewage	7
Burlington	Secondary Effluent	27
Drury Lane Plant	Raw Sewage	12
Burlington	Secondary Effluent	18

The experimental procedures followed for an individual jar test were:

- (i) collect 20-litre grab samples of the influent and effluent streams of the plant and divide each into six, one-litre beaker aliquots;
- (ii) add coagulants in the desired concentration and flash mix the beaker contents at 100 rpm for five minutes;
- (iii) floculate at 40 rpm for 10 minutes if polymer was required, it was added at this stage;
 - (iv) allow samples to settle for 90 minutes at 10 rpm; and
 - (v) discontinue stirring, allowing samples to settle for five minutes and decant supernatant from each beaker for subsequent analyses.

These experimental conditions were followed in an attempt to simulate, as closely as possible, the hydraulic behaviour of a full scale phosphorus removal system. Total phosphorus was measured on all jar test supernatants.

2.6 Analytical Procedures

A summary of the parameters measured during the field study is presented in Table 4.

TABLE 4. PARAMETERS EVALUATED DURING STUDY

```
Raw sewage and primary and secondary effluent ($1, $2, $3).

total phosphorus
NTA
total BODs
suspended solids
volatile suspended solids
soluble total organic carbon
heavy metals (AI, Fe, Zn, Cu, Ni)
temperature
pH
dissolved oxygen
alkalinity
hardness
wastewater flow

Aeration tank contents ($4)

mixed liquor suspended solids
mixed liquor volatile suspended solids
sludge volume index
temperature
dissolved oxygen
return activated sludge flow rate

Waste activated and raw primary sludge and digester supernatant ($5, $6, $7)

total suspended solids
total volatile suspended solids
iron
aluminum
total phosphorus
waste activated sludge flow rate
raw primary sludge flow rate
digester supernatant flow rate
```

Suspended and total solids, pH and total alkalinity analyses were conducted in accordance with Standard Methods (1971). Total organic carbon analyses were done on a Beckman Carbon Analyzer. Calcium and total hardness were determined by the EDTA titrimetric method described in Standard Methods (1971) with magnesium calculated from the difference between total hardness and calcium concentrations. Phosphorus analyses were made by the persuiphate digestion and automated ascorbic acid method.

Samples taken for NTA analyses were preserved immediately with formaldehyde and filtered as soon as possible through a 0.45 micron

membrane filter. They were then analysed by the polarographic method described by Traversy (1971) which is based on the formation of a bismuth-NTA complex at pH 2.0 \pm 0.05. The resultant complex was analysed with a differential cathode ray polarograph.

Samples taken for metal analyses (Fe, Cu, Zn and Ni) were digested with concentrated sulphuric and nitric acid (5 mL of each per 250 mL sample) to dryness. The sample was then made up to 250 mL again with deionized water. If any colour was evident, digestion was repeated using an additional 5 mL of nitric acid. Subsequently, the sample was made up to one litre and heavy metals were determined using the atomic absorption procedures described by Traversy (1971).

2.7 Statistical Treatment of Data

Since the primary objective of the project was to assess the various effects of NTA on the treatment plant, it was necessary to find out by how much different groups of data differed, and how statistically significant this difference was. A simple, but effective, analysis of variance technique was used.

For example, the difference between two populations (group) means was estimated by the sample means with a confidence interval as follows:

$$\mu_1 - \mu_2 = \bar{X}_1 - \bar{X}_2 \pm t_{0.025} S_p \sqrt{\frac{1}{N_1} + \frac{1}{N_2}}$$

where:

 μ_1 = population mean of group 1.

 μ_2 = population mean of group 2.

 \bar{X}_1 = sample mean of group 1.

 \bar{X}_2 = sample mean of group 2.

 N_1 = size of sample group 1.

 N_2 = size of sample group 2.

t_{0.025} = the two-tail "student t" distribution for 95% confidence level.

The "pooled" or combined variance is defined as S_p^2 of all (in this case, two) sample groups considered (Wannacott & Wannacott, 1969).

If
$$\mu_1$$
 - μ_2 includes zero, that is, $t_{0.025} \, ^{\text{S}}_{\text{p}} \sqrt{\frac{1}{N_1} + \frac{1}{N_2}}$ is

greater than \bar{X}_1 - \bar{X}_2 , then the difference is not statistically significant at the 95% confidence level.

When no statistically significant difference is observed between groups, there are the following possible interpretations:

- (1) There is no true difference between the populations from which the samples are taken.
- (2) The variations of sample values within each group are large. This could be caused by sampling errors, size of samples, etc.
- (3) Within group variations are large but there is no true difference.
- (4) There is a true difference but it cannot be observed because of the large variations within each group.

In the analysis of raw data, daily averages were used. Since the treatment plant has an inherent retention time, the effect of time lag must be taken into account when analyzing influent and effluent data. The plant was designed with two aeration tanks in parallel for 1 362 m³/d (0.3 MIGD), giving a retention time of eight hours. Throughout this study, the flow rate was approximately half of the design flow and only one aeration tank was used. The retention time for the active plant was approximately 10 hours. In cases where three or four daily composite samples were taken, the time lag effect was reduced by shifting the effluent samples down one composite sample.

3 RESULTS AND DISCUSSION

3.1 Wastewater Characteristics and Treatment Plant Operation

In Table 5 average values for the raw wastewater to the Waterdown Water Pollution Control Plant are summarized. This provides a reference for the assessment of the treatment plant behaviour under various conditions of stress imposed in the course of this study. The wastewater is typical of a weak municipal wastewater from a low density residential area. The background NTA and phosphorus loadings in the raw wastewater averaged 2.5 mg/L and 6.0 mg/L, respectively. Experimental period averages of the phosphorus and NTA loadings during periods when there was no NTA addition, are presented in Table 6. The daily average variation (Figure 3) and diurnal variations (Figures 4 and 5) of the phosphorus and NTA are also presented. No correlation between NTA/phosphorus loading and the day of the week was observed. Diurnal variation studies of the NTA loading showed peak values of approximately 12 mg/L in the afternoon and minimum values of 0.1 mg/L at night.

The experimental period averages of the secondary effluent characteristics are tabulated in Table 7 and the operational parameters in Table 8. Although the plant was designed as a conventional activated sludge system, the F/M ratio and percent return sludge suggest the plant operation could be more closely approximated by an extended aeration process. As summarized in Table 7, during most of the experimental periods an effluent of acceptable quality was produced.

3.2 Impact of NTA on Treatment Plant Operation

3.2.1 General

Laboratory studies by Thompson and Duthie (1968) have shown that the sedimentation rate and the sludge volume index of raw municipal wastewater are not affected by the addition of up to 50 mg/L of NTA. Field studies by Shumate et al (1970) also showed that NTA had no observable, adverse effects on the overall BOD_5 removal efficiency of an activated sludge plant. Dosages of up to 16 mg/L of NTA were used in the study.

TABLE 5. RAW SEWAGE CHARACTERISTICS

Experimental Periods Parameters	Jan I	(A-0), 1972 to 1, 1972	Jan 2	0+Alum 4, 1972 to 1, 1972	Feb 1	8+Alum 4, 1972 to 6, 1972	Mar	6+A1um 7, 1972 0	NTA-0+ Apr 11, to Apr 28,	1972	NTA-8+ May 1, to May 11,	1972	NTA-16+FeC May 15, 19 to May 26, 19	72 Ma	y 30 t	A-0 , 1972 o , 1972	Nov	TA-0 6, 1972 to 9, 1972	Dec	7A-8 4, 1972 10 7, 1972	Jan	A-16 1, 1973 to 4, 1973	Jan 2	TA-0 9, 1973 to 1, 1973	Feb 2	rA-8 6, 1973 to 1, 1973	Mar 2	(A-16 26, 1973 to 5, 1973
BOD ₅	132*	(97)**	69	(21)	76	(26)	99	(40)	62	(30)	80	(34)	59 (24)	52	(20)	98*	(30)**	109	(82)	102	(37)	121	(55)	119	(35)	60	(12)
TOC	24	(10)	83	(57)	73	(23)	53	(12)	27	(12)	26	(16)	40	(9)	27	(12)	33	(19)	40	(12)	28	(10)	52	(22)	47	(21)	27	(6)
Phosphorus	7.3	(2.9)	7.4	(4.0)	7.1	(1.4)	5.8	(3.3)	3.4	(1.9)	5.1	(3.0)	8.3 (4	.3) 6	8.8	(3.9)		T:		-		•				<u>.</u>		
Suspended Solids	105	(47)	131	(30)	122	(29)	106	(26)	135	(27)	198	(49)	204 (65) 1	144	(43)	117	(94)	57	(28)	94	(40)	153	(59)	133	(34)	93	(27)
Volatile Suspended Solids	84	(42)	114	(26)	106	(26)	90	(25)	86	(19)	119	(23)	146 (36) 1	117	(32)	84	(51)	47	(25)	79	(39)	118	(34)	113	(33)	68	(20)
Al	0.168	(0.040)	0.177	(0.140)	0.181	(0.140)	0.258	(1.410)	0.175 (0.152)	0.160 (0.162)	0.110 (0.0	75) 0.2	221	(0.197)	0.165	(0.156)	0.153	(0.118)	0.174	(0.079)	0.204	(0.112)	0.242	(0.139)	0.214	(0.179)
Cu	0.076	(0.016)	0.048	(0.02)	0.067	(0.015)	0.057	(0.016)	0.074 (0.026)	0.086 (0.018)	0.103 (0.0	45) 0.0	86	(0.025)	0.119	(0.038)	0.070	(0.023)	0.084	(0.029)	0.091	(0.035)	0.078	(0.035)	0.038	(0.017)
Fe	1.10	(1.48)	0.49	(0.07)	0.54	(0.18)	0.62	(0.22)	9.18	(6.41)	18.4	(8.74)	21.6 (12	.5) 3.	03	(4.66)	0.546	(0.272)	0.274	(0.226)	0.352	(0.219)	0.411	(0.263)	0.506	(0.334)	0.389	(0.678)
Zn	0.137	(0.080)	0.173	(0.018)	0.159	(0.040)	0.077	(0.040)	0.161 (0.063)	0.156 (0.066)	0.169 (0.0	73) 0.1	194	(0.062)	0.256	(0.087)	0.157	(0.055)	0.189	(0.131)	0.198	(0.100)	0.176	(0.073)	0.111	(0.095)
NI	0.003	(0.002)	0.004	(0.001)	0.009	(0.007)	0.016	(0.003)	0.014 (0.002)	0.017 (0.004)	0.024 (0.0	24) 0.0)15	(0.002)		*		18 4		Ř				u .		5(4))
Pb			0.030	(0.014)	0.037	(0.027)	0.017	(0.008)	0.027 (0.023)	0.024 (0.008)	0.028 (0.0	09) 0.0	27	(0.006)		A STATE OF THE STA						•		2		200
Alkalinity				n .					246	(69)	327	(11)	381	33)) 	562	(50)	37C	(45)	341	(29)	343	(33)	335	(37)	289	(26)
Hardness				•		8				-		•/			-		278	(88)	471	(176)	469	(85)	403	(100)	270	(67)	415	(89)
Flow	0.008	(0.010)	0.077	(0.015)	0.086	(0.032)	0.131	(0.043)	0.182 (0.050)	0.172 (0.030)	0.106 (0.0	30) 0.1	103	(0.020)		2				2	0.100	(0.020)	0.117	(0.060)	0.220	(0.030)
рН	7.6	(0.8)	8.0	(0.2)	7.8	(0.1)	7.7	(0.2)	7.5	(0.3)	7.5	(0.6)	8.0 (0	.2) 7	7.8	(0.1)	7.9	(0.2)	8.0	(0.1)	7.9	(0.2)	7.9	(0.2)	8.0	(0.1)	7.9	(0.2)

 $^{^{\}pm}$ All parameters in mg/L except pH and flow (flow expressed in MGD). $^{\pm\pm}$ Standard deviations in parentheses.

TABLE 6. NTA AND PHOSPHORUS CONCENTRATIONS IN RAW SEWAGE

Experimental Period	H, NTA mg/L	Phosphorus mg/L
Jan 10 - Jan 21, 1972	2.87 (2.24)	7.3 (2.9)
Jan 24 - Feb 11, 1972	2.77 (2.78)	7.4 (4)
Apr 11 - Apr 28, 1972	1.16 (1.19)	3.4 (1.9)
May 30 - June 7, 1972	2.20 (2.16)	6.8 (3.9)
Nov 6 - Nov 19, 1972	1.98 (2.64)	
Jan 29 - Feb 11, 1973	2.55 (6.55)	

^{*} Standard deviations in parentheses.

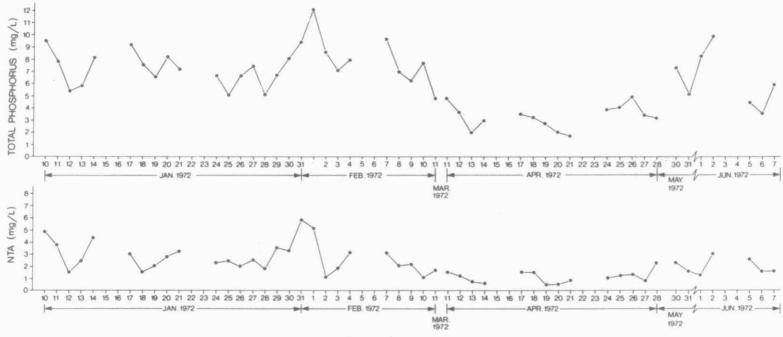


FIGURE 3. DAILY TOTAL PHOSPHORUS AND NTA VARIATION IN WATERDOWN RAW WASTEWATER

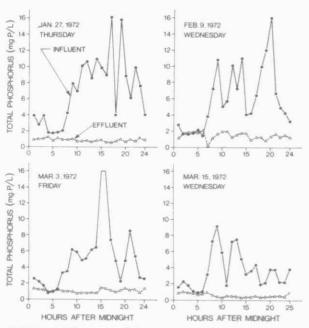


FIGURE 4. DIURNAL VARIATION OF TOTAL PHOSPHORUS IN RAW WASTEWATER AND SECONDARY EFFLUENT

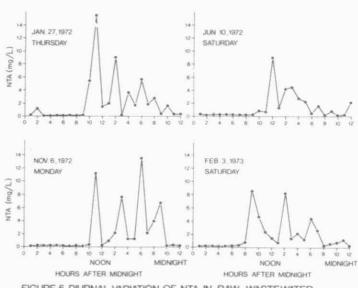


FIGURE 5. DIURNAL VARIATION OF NTA IN RAW WASTEWATER

TABLE 7. SECONDARY EFFLUENT CHARACTERISTICS

Experimental Periods Parameters	Jan 1	TA-0 0, 1972 to • 1, 1972	Jan 2	0+A1um 24, 1972 to 1, 1972	Feb 1	8+Alum 4, 1972 to 6, 1972	Mar	16+Alum 7, 1972 to 4, 1972	NTA-0 Apr 11 t Apr 28	0	NTA-8- May I to May II	1972	t	+FeCl, , 1972 o , 1972	May 30	TA-0 1, 1972 to 7, 1972	Nov	TA-0 6, 1972 to 9, 1972	Dec	TA-8 4, 1972 to 7, 1972	Jan	7A-16 1, 1973 to 14, 1973	Jan 2	TA-0 9, 1973 to 1, 1973	Feb 2	TA-8 6, 1973 to 1, 1973	Mar 2	ra-16 26, 1973 to 5, 1973
800,	20*	(24)**	9	(6)	11	(4)	17	(7)	6	(3)	9	(7)	12	(4)	9	(6)	22*	(12)**	24	(9)	33	(10)	11	(9)	12	(4)	10	(3)
тос	3.1	(6.0)	31.0	(18)	28.3	(9.1)	28.6	(9.7)	18	(9)	14	(4)	15	(1)	12	(6)	21	(7)	38	(23)	22	(5)	19	(4)	24	(5)	19	(2)
Phosphorus	8.3	(1.5)	1.9	(1.1)	1.5	(0.6)	0.96	(0.30)	0.5	(0.2)	0.5	(0.1)	0.8	(0.5)	3.0	(1.8)		-		NGS		i i						
Suspended Solids	48	(75)	22	(9)	29	(7)	25	(3)	9	(3)	10	(3)	14	(9)	12	. (9)	15	(5)	10	(6)	10	(4)	10	(5)	13	(6)	9	(3)
Volatile Suspended Solids	12	(7)	16	(5)	22	(6)	20	(3)	7	(4)	8	(3)	12	(9)	10	(8)	13	(5)	8	(5)	3	(3)	8	(3)	10	(5)	7	(2)
Al	0.136	(0.074)	0.658	(0.450)	0.717	(0.587)			0.063	(0.030)	0.342	(0.011)	0.028	(0.007)	0.111	(0.085)	0.075	(0.064)	0.105	(0.078)	0.118	(0.066)	0.072	(0.044)	0.120	(0.081)	0.120	(0.046)
Cu	0.037	(0.008)	0.015	(0.007)	0.023	(0.007)	0.025	(0.005)	0.021	(0.006)	0.015	(0.003)	0.015	(0.002)	0.025	(0.007)	0.064	(0.039)	0.031	(0.025)	0.038	(0.019)	0.025	(0.030)	0.030	(0.028)	0.025	(0.014)
Fe	0.275	(0.350)	0.173	(0.080)	0.275	(0.100)	0.325	(0.130)	0.630	(0.347)	0.700	(0.394)	0.670	(0.279)	0.340	(0.450)	0.324	(0.124)	0.083	(0.046)	0.167	(0.077)	0.095	(0.057)	0.249	(0.218)	0.134	(0.123)
Zn	0.063	(0.056)	0.110	(0.037)	0.138	(0.040)	0.129	(0.130)	0.058	(0.020)	0.098	(0.037)	0.113	(0.062)	0.081	(0.015)	0.182	(0.055)	0.128	(0.022)	0.179	(0.122)	0.097	(0.039)	0.174	(0.030)	0.077	(0.054)
MI	0.002	(0.002)	0.003	(0.001)	0.009	(0.008)	0.014	(0.003)	0.009	(0.002)	0.010	(0.002)	0.009	(0.002)	0.009	(0.003)		4		120		e e		2		2		1
РЬ	0.005	(0.003)	0.013	(0.0180)	0.0184	(0.018)	0.010	(0.008)	0.005	(0.002)	0.005	(0.005)	0.005	(0.002)	0.005	(0.001)				2				(8)		4		**
pH	7.0	(0.1)	7.0	(0.1)	7.0	(0.1)	7.1	(0.1)	7.2	(0.1)	7.0	(0.2)	7.2	(0.1)	7.1	(0.1)	7.4	(0.1)	7.0	(1.4)	7.2	(1.3)	7.4	(0.1)	7.4	(0.1)	7.5	(0.1)

TABLE 8. WATERDOWN POLLUTION CONTROL PLANT OPERATIONAL CHARACTERISTICS

Experimental Periods	800 Removal	र Return Sludge	Aeration Time h	MLSS mg/L	MLVSS mg/L	Temp. °C	F M	DO mg/L	SVI
NTA-0, Jan 10-21, 1972	87	33	9.8	2 089	1 792	11	0.13	2.4	123
NTA-0+Alum, Jan 24 - Feb 11, 1972	85	89	7.9	2 756	1 963	9	0.06	2.3	165
NTA-8+Alum, Feb 14 - Mar 6, 1972	86	97	6.8	2 866	1 912	10	0.08	3.2	228
NTA-16+Alum, Mar 7-24, 1972	80	74	5.1	3 691	2 488	9	0.10	5.7	161
NTA-0+FeCl ₃ , Apr 11-28, 1972	88	67	3.8	2 709	1 997	11	0.12	6.0	109
NTA-8+FeCl ₃ , May 1-11, 1972	86	69	4.0	2 861	2 098	12	0.14	4.1	161
NTA-16+FeCl ₃ , May 15-26, 1972	79	33	8.2	2 474	1 836	15	0.07	2.7	125
NTA-0, May 30 - June 7, 1972	84	88	5.9	2 623	1 927	16	0.06	1.0	114
NTA-0, Nov 6-19, 1972	75	34	(<u>=</u>)/	1 645	1 350	15		3.6	578
NTA-8, Dec 4-17, 1972	73	28		1 484	1 169	12		4.4	516
NTA-16, Jan 1-14, 1973	66	30	0ece2	1 463	1 169	10	960	4.9	498
NTA-0, Jan 29 - Feb 11, 1973	92	29	8.6	1 477	1 216	10	0.21	4.8	667
NTA-8, Feb 26 - Mar 11, 1973	88	53	6.4	2 324	1 861	10	0.16	4.1	394
NTA-16, Mar 26 - Apr 5, 1973	82	42	3.7	2 212	1 579	10	0.17	3.8	167

^{*} All parameters in mg/L except pH. ** Standard deviations in parentheses.

3.2.2 Treatment plant efficiency

The BOD_5 and suspended solids removal efficiencies in this study were monitored routinely during all the experimental periods. Table 9 shows that in each period the percent BOD_5 removal decreased slightly with increasing NTA level. The fact that this trend was evident in all four periods leads to the hypothesis that high levels of NTA do have some adverse effect on plant operation. To test this hypothesis, the data in Table 9 are grouped into the three NTA levels and an analysis of variance carried out. The statistical data presented in Table 10 shows that there was a slight, but statistically significant, decrease in BOD_5 removal at an NTA level of 16 mg/L.

TABLE 9. INFLUENT NTA CONCENTRATIONS AND
TREATMENT PLANT BOD5 AND SUSPENDED
SOLIDS REMOVAL FOR EACH EXPERIMENTAL
PERIOD

	LEITTOD			
Experimental Periods	Date	NTA Level	% BOD ₅ Removal	% Suspended Solids Remova
NTA-0 No Alum	Jan 10 - Jan 21	3.0	86.5 (6.7)*	85.2 (3.2)
NTA-0 A1 um	Jan 24 - Feb 11	2.7	84.4 (9.4)	83.1 (5.6)
NTA-8 ATum	Feb 14 - Mar 6	6.1	85.3 (5.6)	73.8 (10.0)
NTA-16 Alum	Mar 7 - Mar 24	14.5	79.9 (9.8)	74.5 (8.4)
NTA-O FeCl ₃	Apr 11 - Apr 28	1.2	88.0 (9.9)	93.0 (3.1)
NTA-8 FeCl ₃	May 1 - May 4	10.8	86.4 (11.0)	94.6 (1.9)
NTA-16 FeCl;	May 15 - May 26	19.0	78.9 (8.4)	92.8 (4.8)
NTA-0	May 30 - June 7	2.3	83.5 (7.8)	91.9 (3.9)
NTA-0	Nov 6 - Nov 19	2.0	74.9 (16.2)	80.8 (11.8)
NTA-8	Dec 4 - Dec 17	9.5	73.0 (11.8)	81.3 (10.5)
NTA-16	Jan 1 - Jan 14 1973	21.9	65.5 (10.6)	88.9 (2.6)
NTA-0	Jan 29 - Feb 11	2.6	91.5 (3.9)	92.6 (6.0)
NTA-8	Feb 26 - Mar 11	7.5	87.6 (6.2)	89.4 (4.9)
NTA-16	Mar 26 - Apr 5	9.0	81.8 (7.3)	89.1 (3.5)

^{*} Standard deviations in parentheses.

TABLE 10. EFFECT OF NTA LOADING ON BOD5 AND SUSPENDED SOLIDS REMOVAL

	Average	Average		% BOD ₅ Rem	ova!	₹ Suspended Solids Removal			
	mg/L H ₃ NTA	Temp °C	n	Average	Standard Deviation	n	Average	Standard Deviation	
μ	2.3	11.5	69	85.2	10.3	69	87.7	7.7	
11.2	8.3	10.8	40	82.9	10.4	41	83.4	11.5	
ii 3	16.0	10.6	43	78. 1	11.0	42	84.7	10.6	
	μ1 - μ2			2.3 ± 4.0	NS		4.3 ± 3.6	s	
	UE = UE =			7.1 ± 4.1	S	3.0 ± 3.4 NS			
	U2 - U3	a		4.8 ± 4.7	NS	-0.7 ± 4.8 NS			

NS = Not significant at the 95% confidence level. S = Significant at the 95% confidence level.

3.3 Effect of NTA on Chemical Precipitation of Phosphorous

3.3.1 General

When ferric or aluminum salts are added to wastewater containing phosphates, the metals react with the phosphate anions to precipitate phosphorus. In the presence of NTA, one would expect the chelating capacity of the NTA to compete with the phosphate anions for the metallic ions, thereby resulting in a reduced phosphorus removal efficiency.

Forsberg and Wiberg (1968) treated sewage effluent with 100 mg/L of aluminum sulphate in the presence of 0, 1, 5 and 10 mg/L of NTA. The phosphorus removal efficiency was observed to decrease by about 10% between 0 and 10 mg/L NTA. However, the statistical significance of the results was not clear. Bouveng et al (1968) also reported qualitative results indicating a slight decrease in phosphorus removal with alum at NTA levels from 0 to 10 mg/L. Gudernatsch (1970) reported that the influence of NTA on phosphorus removal with lime was insignificant and the results with ferric and alum salts were similar to Bouveng's. The NTA concentration under study was 25 mg/L as Na₃NTA·H₂O.

3.3.2 Bench scale studies

To elucidate on the above observations, a series of jar tests were carried out in the laboratory using domestic sewage with different levels of NTA and chemical precipitants. The results are presented in Tables 11 and 12. An analysis of variance on the

experimental data showed that the presence of up to 16 mg/L of NTA had no effect on chemical precipitation of phosphorus when using alum or ferric chloride.

TABLE 11. BENCH SCALE EVALUATION OF THE IMPACT OF NTA ON PHOSPHORUS REMOVAL IN RAW SEWAGE

		Mean Percent Removal As A Function of NTA And Coagulant Dosage						age	Analysis Of Variance						
Coagulant	Replicates	NTA Coagulant Dosage mg/L							Source of Variation	Sum of	Degrees of	Hean	Fe	r	
		mg/L	0.0	5.0	10.0	15.0	20.0	30.0	Source of Variation	Squares	Freedom	Squares	. c	0.005	
Ferric Chloride	6	0.0	0.0	21.8	39.7	49.5	61.4	73.7	NTA	1 749.0	2	874.5	8.58	3.10	
		8.0	0.0	24.0	34.9	46.9	57.7	72.7	Coagulant Dosage	70 819.0	5	14 163.8	139.00	2.33	
	1 -	16.0	0.0	22.5	50.8	62.6	70.9	84.9	NTA x Coagulant Dosage	1 019.0	10	101.9	1.12	1.95	
									Error	8 159.0	90	90.7			
A I um	6	0.0	0.0	38.5	64.1	80.7	88.9	93.1	NTA	187.6	2	93.8	1,22	3.10	
		8.0	0.0	47.9	63.5	76.0	86.1	91.8	Coagulant Dosage	109 903.0	5	21 980.6	285.41	2.33	
		16.0	0.0	44.6	75.0	83.0	86.7	92.9	NTA x Coagulant Dosage	770.1	10	77.0	1.02	1.95	
									Error	6 779.9	90	75.3			

TABLE 12. BENCH SCALE EVALUATION OF THE IMPACT OF NTA ON PHOSPHORUS REMOVAL IN SECONDARY EFFLUENT

		Mean Percent Removal As A Function Of NTA And Coagulant Dosage							Analysis Of Variance					
Coagulant	Replicates	NTA	A Coagulant Dosage mg/L						Source of Variation	Sum of	Degrees of	Mean		
		mg/L	0.0	5.0	10.0	15.0	20.0	30.0		Squares	Freedom	Squares	Fc	F0.005
Ferric Chloride	6	0.0	0.0	27.1	44.8	61.1	75.4	84.5	NTA	363.7	2	181.9	9.78	3,90
		8.0	0.0	33.0	52.3	62.0	76.2	85.9	Coagulant Dosage	85 357.3	5	17 071.5	918.14	2.33
		16.0	0.0	28.1	44.6	57.4	70.5	82.8	NTA x Coagulant Dosage	185.9	10	18.6	0.13	1.95
									Error	13 381.2	90	148.7		
ATum	6	0.0	0.0	53.9	75.3	85.4	90.5	93.5	NTA	29.1	2	14.5	2.16	3.09
		8.0	0.0	54.4	75.5	81.6	88.5	92.5	Coagulant Dosage	128 632.8	5	25 726.6	3 821.02	2.32
		16.0	0.0	54.6	74.3	83.9	88.9	91.2	NTA x Coagulant Dosage	67.3	10	6.7	0.15	1.95
									Error	4 850.1	108	44.9		

3.3.3 Field studies

The effect of NTA on the chemical precipitation of phosphorus was also examined at field scale. Table 13 indicates the effect of NTA on primary phosphorus removal by FeCl3. The removal increased by 5.4% between 1.2 and 10.8 mg/L NTA and by 7.3% between 1.2 and 19.2 mg/L NTA. No significant difference was observed between 10.8 and 19.2 mg/L NTA. It should be observed that the slight increases are just barely significant and phosphorus removal has increased rather that decreased.

TABLE 13. EFFECT OF NTA ON PHOSPHORUS REMOVAL WITH FeC13

	Aver NTA L mg/L H	evel	Averag Temp °C	e n	Aver Phosphoru			$\Sigma(x_i-\bar{x})^2$	Standard Deviation
μ1	1.	2	10.5	14	83	.97		633.13	6.9
μ ₂	10.	8	12.0	10	89	. 36		195.56	4.7
μз	19.	2	14.5	11	91	. 26		55.61	2.4
				35				884.30	
		S	$p^2 = \frac{884}{3}$.30 =	27.63		Sp	= 5.26	t _{0.05} = 2.042
и	1 - µ2	=	-5.39 ±	10.74	$\sqrt{\frac{1}{14} + \frac{1}{10}}$	=	-5.3	9 ± 4.4	S
П	1 - µ3	=	-7.29 ±	10.74	$\sqrt{\frac{1}{14} + \frac{1}{11}}$	=	-7.2	9 ± 4.3	S
Ц	2 - µ3	=	-1.9 ±	10.74	$\sqrt{\frac{1}{10} + \frac{1}{11}}$	=	-1.9	± 4.7	NS

NS = Not significant at the 95% confidence level.

S = Significant at the 95% confidence level.

The NTA effect when alum was added to the aeration tank is shown in Table 14. No statistically significant difference was observed. In the field study at Gloucester (Shannon and Kamp, 1973) no quantifiable effect on phosphorus removal was observed with either alum, FeCl₃ or lime in the presence of increased NTA levels. The results obtained from the Waterdown field study confirm the findings in the bench scale jar tests and the published literature. Nitrilotriacetic acid dosages up to 16 mg/L do not adversely affect the chemical precipitation of phosphorus in the activated sludge process.

TABLE 14. EFFECT OF NTA ON PHOSPHORUS REMOVAL WITH ALUM

	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	x Average % Phosphorus Remova	$\sum_{1} \left[\sum_{i} (X_{i} - \overline{X})^{2} \right]$	Standard Deviation
μ1	2.8	9.4	17	73.15	2 476.88	12.4
μ2	6.4	9.7	17	78.57	985.58	7.8
μз	14.0	9.2	16	76.08	3 047.75	14.3
			50		6 510.21	
	Sp	$=\frac{6510.}{47}$	21 =	138.52 S _p	= 11.769	t _{0.05} = 2.0
Ц	1 - µ2 =	-5.42 ± 2	3.66	$\sqrt{\frac{1}{17} + \frac{1}{17}} = -5$.42 ± 8.12	NS
Ц	1 - µ3 =	-2.93 ± 2	3.66	$\sqrt{\frac{1}{17} + \frac{1}{16}} = -2$.93 ± 8.26	NS
1.1	₂ - μ ₃ =	2.49 ± 2	3.66	$\sqrt{\frac{1}{17} + \frac{1}{17}} = 2$.49 ± 8.26	NS

NS = Not significant at the 95% confidence level.

3.4 NTA Degradation in the Treatment Plant

3.4.1 General

Earlier studies have indicated that NTA is biodegradable and is removed by the conventional activated sludge process (Thompson and Duthie, 1968). It has also been reported that NTA does not affect normal treatment plant operation (Shumate et al, 1970). A general impression of NTA removal may be obtained by examining Figure 6 (a), (b) and (c) where typical influent NTA concentrations and the overall amount removed are plotted. Figure 7 (a), (b) and (c) show examples of overall NTA removal (in percent removal) and NTA input concentration. It may be seen from the Figures that the amount of NTA removed follows the input NTA concentration closely and there is a general trend that the removal rate decreases with increasing NTA concentration. That is, the rate of NTA removal by acclimated sludges is a first order reaction. For the case of stoichiometric NTA-metal complexes, Shannon et al (1978) also determined from batch data that biodegradation of these complexes could be approximated by a first order reaction. The overall removal

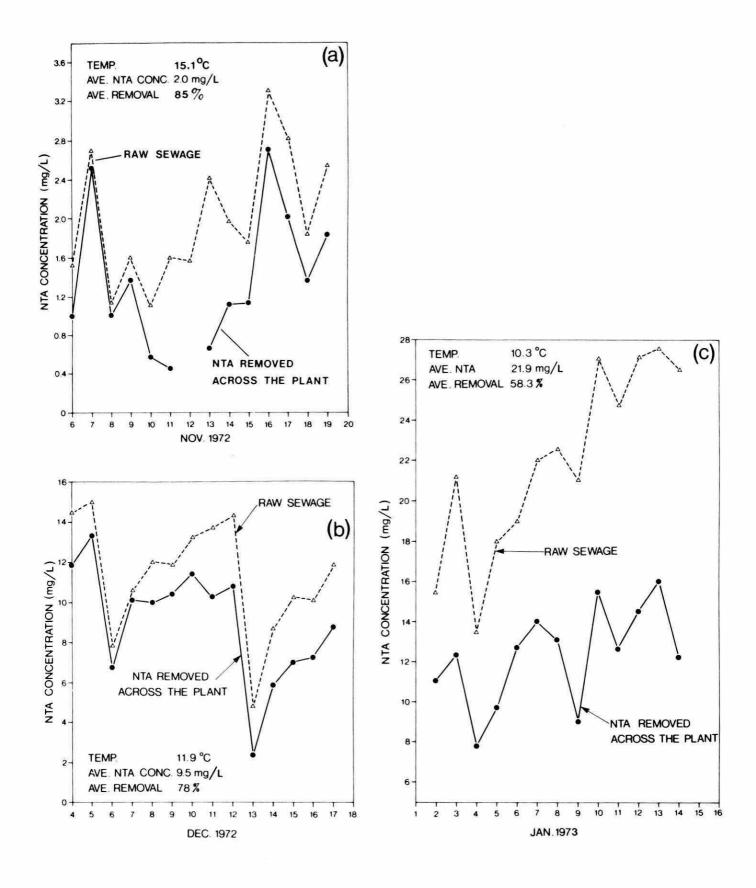


FIGURE 6. NTA DEGRADATION

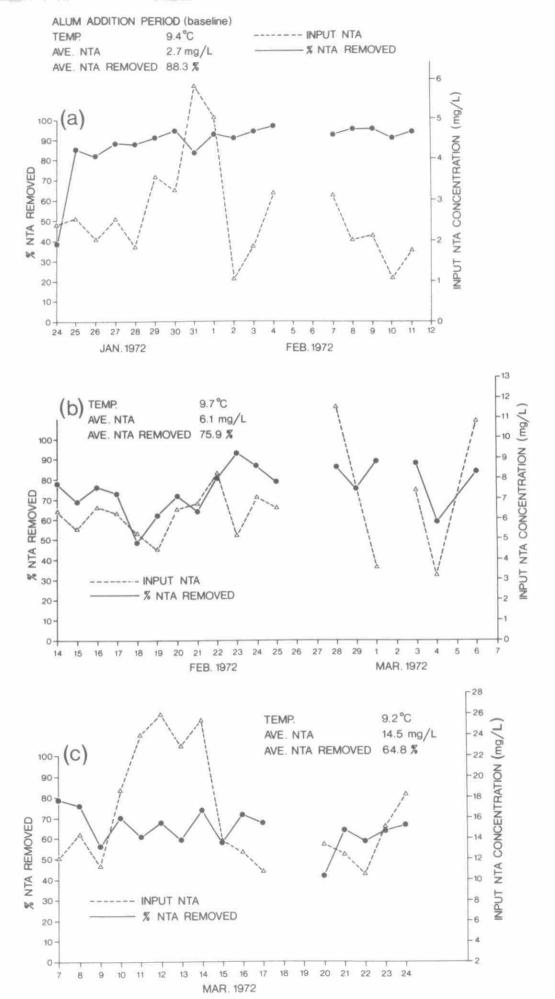


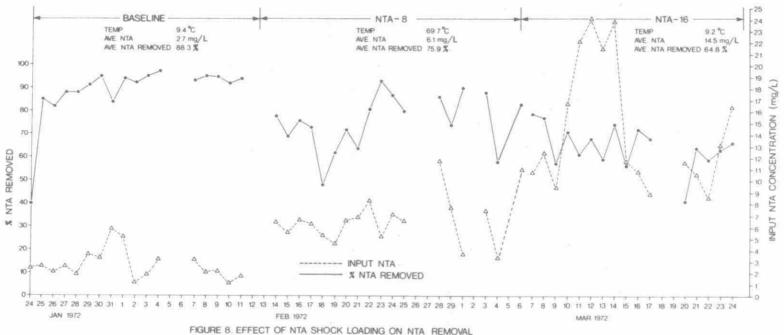
FIGURE 7. PERCENT NTA REMOVAL AS A FUNCTION OF INLET NTA CONCENTRATION

of NTA through the plant (primary settling and activated sludge treatment) has been used throughout the report. This is because data from this investigation and that of others has shown that negligible removal occurs in primary clarification. Thus, the removals shown are attributed to the secondary process only.

3.4.2 Acclimatization of microorganisms to NTA

Spiking synthetic sewage in a laboratory size unit with NTA, Swisher et al (1967) found that two to three weeks acclimation were needed before NTA degradation would proceed smoothly for fresh activated sludge. In separate studies, Bouveng et al (1968) found a 35 to 40-day acclimation period was required with sewage in bench and pilot scale units. In their study, Shumate et al (1970) used five weeks of acclimatization for the 2 mg/L NTA level, one week for the following 8 mg/L NTA addition period, and none for the 16 mg/L NTA period. A significant finding in Shumate's work was that an acclimatization period was not necessary for an increase in NTA dosage from 8 to 16 mg/L, as long as the sewage was already well acclimatized at 8 mg/L. Cleasby et al (1974) found that an increase in NTA feed concentration from 4 to 8 mg/L caused no increase in effluent NTA, but an increase to 16 mg/L did show a higher initial concentration of NTA in the effluent. However, only a few days were required to acclimate the system. Renn (1974), who was studying NTA loading on a package treatment plant, found that a three-month acclimation period was required when the NTA level was shifted from 0.5 to 30 mg/L. Shannon et al (1978) observed that in batch studies of biodegradation of various NTA-metal complexes, acclimation generally occurred in less than seven days. The general conclusion to be drawn from these studies is that an acclimation period is required for sludge which has not been previously exposed to NTA.

Acclimation periods were also observed in this study. During the alum addition period, three NTA dosage levels were studied consecutively without periods of acclimatization in between. As indicated in Figure 8, there was a noticeable drop in NTA removal on February 14 when the NTA level was increased to 6.5 mg/L from a baseline value of



about 2 mg/L. A similar, but not as pronounced a drop may be noted on March 10 when the NTA level increased from 9 to 24 mg/L. During the NTA addition period from January 29, 1973 to March 8, 1973, there was a two-week acclimation period between NTA spiking levels. Even after the two-week acclimation period, there was still some initial reduction in NTA removal as a result of the transition from 2.6 to 7.5 mg/L NTA.

The Waterdown data demonstrated that an acclimation period is required following an increase in NTA loading even when the sludge has been subjected to NTA for prolonged periods. For NTA loading step changes of 5 to 10 mg/L, the acclimation period appears to be approximately three weeks.

3.4.3 Chemical addition

The effect of alum and ferric chloride additions on NTA degradation is demonstrated in Tables 15 and 16. Both chemical additions fail to indicate a statistically significant difference in NTA removal between samples with and without chemical addition. The sets of data were chosen for analyses on the basis of constant temperature and NTA dosage.

TABLE 15. EFFECT OF ALUM ADDITION ON NTA REMOVAL

	Precipitant	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	x Average % NTA Removal	$\Sigma (X_i - \bar{X})^2$	Standard Deviation
μι	No Alum	3.0	11.0	9	78.8	1 788.22	14.9
μ2	With Alum	2.7	9.4	17	88.3	2 798.26	13.2
			k	26		4 586.48	
	S _p ²	$=\frac{4\ 586.48}{24}$			S _p = 13.82	t _{0.05}	= 2.064
	μ1 - μ2	= -9.5 ± 28	$3.53\sqrt{\frac{1}{9}+}$	1 17	= -9.5 ± 11	. 76	NS

NS = Not significant at the 95% confidence level.

TABLE 16. EFFECT OF FeC13 ADDITION ON NTA REMOVAL

	Precipitant	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	X Average % NTA Removal	Σ(X ₁ -X̄) ²	Standard Deviation
ш1	No FeCl ₃	9.5	12,0	14	78.64	1 989.83	12.4
μ2	With FeCl ₃	10.8	12.0	10	68.21	3 425.01	19.5
				24		5 414.84	
	S _p ²	$=\frac{5\ 414.84}{22}$		-	S _p ² = 15.68	t _{0.05}	= 2.074
	111 - 112	= 10.43 ± 3	32.54	+ 1	= 10.43 ±	13.47	NS

NS = Not significant at the 95% confidence level.

3.4.4 Temperature effect

Rudd and Hamilton (1972) found in a model-scaled aerated sewage lagoon that temperatures of 0.5, 5 and 15°C resulted in NTA removal of 25, 47 and 93%, respectively. The NTA input concentration was maintained at 14.0 mg/L. Eden et al (1972) varied the temperature of a laboratory unit containing detergent-free sewage with 5 mg/L NTA from 5 to 20°C and finally to 7.5°C. The corresponding NTA removals were 3, 98 and 82%. Bouveng et al (1968) concluded from a pilot plant study with synthetic sewage that degradation of NTA is more efficient at 20°C than 5°C.

Statistical analysis of the Waterdown data presented in Table 17 shows that there is a significant difference between NTA removal efficiencies at different temperatures. At NTA concentrations of approximately 2.5 mg/L, it is observed that a significant difference in NTA removal exists between 15.5 and 9.4°C. At the high NTA concentrations of about 20 mg/L, there was a very significant difference in NTA removal between 10.3 and 14.5°C. The results show that NTA degradation is significantly affected by wastewater temperature. The magnitude of the temperature effect increases with increased NTA loadings to the treatment plant.

TABLE 17. EFFECT OF TEMPERATURE ON NTA REMOVAL

	Average Temp °C	Average NTA Level mg/L H ₃ NTA	n	X Average ₹ NTA Removal	$\mathbb{E}(\mathbf{x}_{\hat{i}} - \hat{\mathbf{x}})^2$	Standard Deviation
-1	15.5	2.3	7	98.55	11.92	1.4
U 2	9.8	2.6	14	85.29	1 524.95	10.8
113	9.4	2.7	17	88.29	2 798.26	13.2
	-		38		4 335.13	
	5_2 =	4 335.13		S _p = 11.13	t _{0.05}	= 2.03
			± 22	59 √ 1 + 1 14		
		= 13.26		$59\sqrt{\frac{1}{7} + \frac{1}{14}}$ $59\sqrt{\frac{1}{7} + \frac{1}{17}}$	= 13.26	± 10.46

	Average Temp °C	Average NTA Level mg/L H;NTA	n	x̃ Average % NTA Removal	Σ(x ₁ -x̄) ²	Standard Deviation
121	10.3	21.9	13	58.29	970.31	9.0
uz:	14.5	19.2	10	89.36	2 387.44	16.3
			23		3 357.75	
	5 ² =	3 357.75		S _p = 12.35	t _{0.05}	= 2.074
	μ ₁ - μ ₂	= 31.07	± 25	.61 $\sqrt{\frac{1}{13} + \frac{1}{10}}$	= 31.07	± 10.77

NS = Not significant at the 95% confidence level. S = Significant at the 95% confidence level.

3.4.5 Effect of NTA loading

In a study of NTA removal at a 1 $362 \, \mathrm{m}^3/\mathrm{d}$ (0.3 MIGD) sewage treatment plant (Shumate et al, 1970), it was found that NTA removal was 89.4, 90.0 and 75.2% for nominal NTA dosages of 2, 8 and 16 mg/L, respectively. No adverse effect on the treatment plant due to the NTA additions was observed. The Gloucester study (Shannon and Kamp, 1973) reported NTA removal of 73 and 61% at average NTA loadings of 0.7 and 2.2 mg/L, respectively.

Data from the alum addition period and Phase 2 (2nd trial) of this study were selected for analysis of the effect of NTA loading on its removal because the temperature variations in each period were minimal. Table 18 shows that there is a significant difference in NTA removal between high and low NTA spiking levels. Thompson and Duthie (1968) observed that the rate of NTA removal was in all cases a zero order reaction. If the data obtained in this study at 9 to 10°C is lumped and evaluated, pseudo first order kinetics for NTA removal are

suggested. Sufficient data is not available to repeat the analysis in the higher temperature rate. Pseudo first order kinetics, however, are not uncommon in waste treatment systems treating mixed wastes.

TABLE 18. EFFECT OF NTA LOADING ON ITS REMOVAL

	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	Ä Average % NTA Removal	$\Sigma (X_1 - \widetilde{X})^2$	Standard Deviation
μ,	2.7	9.4	17	88.29	2 798.26	13.2
U2	14,5	9,2	16	64.75	1 303.12	9.3
			33		4 101.38	
	$S_p^2 = \frac{4 \cdot 101}{31}$.38		S _p = 11.50	t _{0.05}	= 2.042
	μ1 - μ2 =	23.54	23	49 17 + 12	= 23.54	± 8.18 S

	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	X Average % NTA Removal	Σ(X ₃ -X) ²	Standard Deviation
uı	2.6	9.8	14	85.29	1 524.95	10.8
Иg	7.5	10.2	13	47.96	2 239.19	13.7
			27		3 764.14	
	$s_p^2 = \frac{3.764}{25}$. 14		S _p = 12.27	t _{0.05}	= 2.06
	μ1 - μ2 =	37.33	25	. 28 $\sqrt{\frac{1}{14} + \frac{1}{13}}$	= 37.33	± 9.74 5

S = Significant at the 95% confidence level.

3.5 Transport of Heavy Metals from Treatment Plant

3.5.1 General

Nitrilotriacetic acid is a strong chelating agent. Its anions can form complexes with metallic cations. From the environmental protection point of view, there is serious concern as to the formation of NTA-metal complexes and their subsequent degree of biodegradation in wastewater treatment plants. If the NTA-metal complexes are not readily degraded in the plant, there will be transport through, or washout of, heavy metals from the treatment plant to the receiving streams. Shannon et al (1978) studied the biodegradation of various NTA-metal complexes and concluded that for most complexes in the concentrations that might occur, metal transport, even at low winter temperatures, is a problem of little practical significance. In the Waterdown study, the net effect of chelate formation and their biodegradation was assessed indirectly by comparing the heavy metal removal efficiencies for periods of different NTA dosages. A reduced heavy metal removal efficiency in the presence of NTA would be indicative of heavy metal transport.

3.5.2 Heavy metal removal and NTA degradation

In a study by Chau and Shiomi (1972), heavy metals such as Cu, Zn, Fe and Ni, were released from Lake Ontario sediment samples in the form of soluble NTA-metal complexes after NTA was added. It was found that once the degradation of the added NTA was completed, the concentrations of the soluble or released metals decreased drastically. Chau and Shiomi (1972) suggested that when the NTA complexing mechanism was destroyed through NTA degradation, all the released metals either recombined with their original anions to form precipitants, or were adsorbed by the sediment. Figure 9 shows the relationship between heavy metal removal efficiencies and percent NTA removal in this study. The heavy metals examined included Zn, Cu, Fe and Al. Except for Cu, the general trend indicates that higher NTA removal generally results in higher heavy metal removal. This is to be expected since a higher NTA removal efficiency means less NTA available to form chelates with the heavy metals. Furthermore, the overall NTA removal may also include the biodegradation of the NTA portion of a NTA-metal complex.

3.5.3 Effect of chemical addition

At relatively constant temperature and NTA loading, the addition of FeCl₃ to the treatment plant was found to improve the removal efficiencies for Zn, Cu and Al by approximately 25%. The results are statistically significant as shown in Table 19. The amount of complexing or solubilization of the heavy metals by NTA may have been reduced as a result of FeCl₃ addition. The iron would compete with the other heavy metals for NTA's chelating capacity with the result that a lesser amount of the other heavy metals are solubilized (Ashforth and Calvin, 1973).

The heavy metal data during the alum addition period was erratic. The main cause of the variation may have been the start-up difficulties. Solids carryover was also observed. As a result, the effect of alum addition could not be assessed.

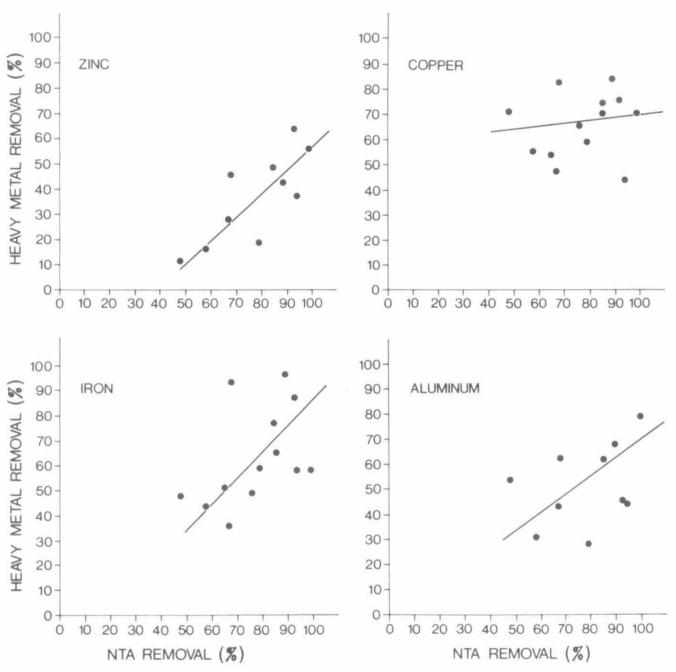


FIGURE 9. HEAVY METAL REMOVAL AS A FUNCTION OF NTA REMOVAL

TABLE 19. EFFECT OF FeC13 ADDITION ON HEAVY METAL REMOVAL IN THE PRESENCE OF NTA

			Zinc	Rem	ova l		
	Precipitant	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	x Average % Heavy Metal Removal	$\Sigma(x_1-\bar{x})^2$	Standard Deviation
μ1	With FeCl ₃	10.8	12.0	7	45.7	1 594.15	16.3
μ2	No FeCl ₃	9.5	11.9	12	19.3	1 377.55	11.2
	<u> </u>			19		2 971.70	
	S _p ²	$= \frac{2971.70}{17}$		•	s _p = 13.22	^t 0.05	= 2.110
	μ1	- μ ₂ = 20	6.4 ± 27.8	$9\sqrt{\frac{1}{7}}$	$+\frac{1}{12} = 26.4 \pm$	13.27	S

		Copper Removal											
	Precipitant	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	X Average % Heavy Metal Removal	$\Sigma(x_i-\bar{x})^2$	Standard Deviation						
μι	With FeCl ₃	10.8	12.0	9	82.06	426.08	7.3						
μ2	No FeCl ₃	9.5	11.9	14	58.70	5 051.87	19.7						
		-L	1	23		5 477.95							
	S _p ²	= ^{5 477.95}			S _p = 16.15	^t 0.05	= 2.08						
	u,	- μ ₂ = 2	3.36 ± 33.	59 J	$\frac{1}{0} + \frac{1}{1/h} = 23.36$	± 14.35	s						

			Alumin	um Re	emoval		
	Precipitant	Average NTA Level mg/L H ₃ NTA	Average Temp °C	n	x Average % Heavy Metal Removal	$\Sigma(X_{i}^{-\bar{X}})^{2}$	Standard Deviation
μ1	With FeCl ₃	10.8	12.0	7	62.6	5 426.03	30.0
μ2	No FeCl ₃	9.5	11.9	12	28.8	4 716.50	20.7
				19		10 142.53	
	S _p ²	$= \frac{10 \ 142.53}{17}$		-	s _p = 24.43	^t 0.05	= 2.11
	μι	- μ ₂ = 3	3.8 ± 51.5	$4\sqrt{\frac{1}{7}}$	$\frac{1}{12} = 33.8$	± 24.51	s

S = Significant at the 95% confidence level.

3.5.4 Effect of NTA loading

Nilsson (1971) added heavy metals and NTA to wastewater samples and found that Cu and Pb escaped precipitation in the presence of NTA ranging in concentrations from 0 to 12 mg/L. Sanchez and Lee (1973) added NTA to dredged lake sediment samples and reported that increasing amounts of Fe and Mg were solubilized due to an increase in NTA concentrations. However, contrary to what would be expected, the copper released from the sediments was found to decrease with increasing NTA. Shannon and Kamp (1973) reported no transport of Fe, Cu, Zn and Pb due to NTA increase. This could be due to the relatively low average NTA dosage used in their study, but there was some evidence of transport of Ni and Cd. The average fraction of metal transported through a full scale waste treatment plant and a pilot plant spiked with NTA operating in parallel, was examined by Cleasby et al (1974). Statistical analysis of the data revealed no significant difference in metal transport between the two plants and no correlation between NTA loading and metal transport. Renn (1974), who examined a small package plant, found that zinc and iron transport increased with increased NTA loading. To evaluate the effect of different NTA effluent levels on heavy metal removal, data from three study periods (iron addition period, second year Phase I and Phase II) were analyzed for significant differences in metal removal. For the metals considered (Al, Zn, Cu, Fe, Ni and Pb) no general trend in heavy metal removal efficiency is evident (Table 20). The removal of aluminum, nickel and lead is independent of NTA concentration. Zinc and iron removal tend to decrease with increasing NTA level, however, in most cases the differences in removal are not statistically significant. For copper and iron removal, the trend is for increased copper and iron removal with increased NTA level in the effluent. However, as with zinc, the differences in removal are, in most instances, not statistically significant. Aluminum, Ni and Pb remova; did not increase with increased NTA level in the effluent. The data, in general, tend to confirm the results obtained by Cleasby et al (1974). That is: zinc transport increased with increased NTA level in the effluent.

TABLE 20. EFFECT OF NTA LOADING ON HEAVY METAL REMOVAL

	NTA		Pe	rcent Me	tal Remo	val	
	Conc. mg/L	Al	Zn	Cu	Fe	Ni	Pb
иı	0.1	79.5	55.6	69.9	58.5	43.5	75.1
L 2	0.1	46.2	64.5	75.3	86.6	38.7	71.8
из	2.0	69.1	42.9	83.7	95.7	19.6	81.7
Li a	3.5	62.6	45.7	82.1	93.3	38.0	75.8
u ₁ - u ₂		NS	NS	NS	s	NS	NS
μ1 - μ1		NS	NS	S	s	S	NS
u1 - u4		NS	NS	s	s	NS	NS
μ2 - μ3		NS	s	s	NS	NS	NS
μ2 - μ4		NS	S	S	NS	NS	NS
μ, - μ,		NS	NS	NS	NS	NS	NS
μ1	0.7	43.8	28.1	47.3	36.2		
μ_2	2.0	28.8	19.3	58.7	58.9		
u ₃	9.1	31.3	15.9	55.0	43.9		
μ ₁ - μ ₂		s	NS	s	s		1
µ1 - µ3		NS	NS	NS	NS		
μ2 - μ3		NS	NS	NS	NS		
μ	0.3	62.7	48.2	70.3	75.9		I
µ2	0.6	45.2	36.4	43.6	58.2	1	
u,	3.9	53.7	11.4	71.4	47.9		
μ ₁ - μ ₂		s	NS	S	s		
µ1 - µ3		NS	s	NS	S		
u ₂ - u ₃		NS	s	s	NS		

NS = Not significant at the 95% confidence level. S = Significant at the 95% confidence level.

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